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## Magnetostrictive and elastic properties of $\text{Fe}_{100-x}\text{Mox}$ (2

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# Magnetostrictive and elastic properties of $\text{Fe}_{100-x}\text{Mo}_x$ ( $2 < x < 12$ ) single crystals

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In this paper we investigate the magnetostrictive  $[(3/2)\lambda_{100}$  and  $(3/2)\lambda_{111}]$  and elastic ( $c'$  and  $c_{44}$ ) behavior of single crystalline alloys  $\text{Fe}_{100-x}\text{Mo}_x$  for  $2 < x < 12$ ; the magnetoelastic coupling constants ( $-b_1$  and  $-b_2$ ) are computed from the measurements. Similar to other Fe-X ( $X=\text{Al}$ , Ga, and Ge) alloys, the tetragonal magnetostriction  $(3/2)\lambda_{100}$  increases monotonically from  $\sim 70 \times 10^{-6}$  at  $\sim 2.5$  at. % Mo to a maximum of either  $\sim 100 \times 10^{-6}$  at  $\sim 8$  at. % Mo for the slow cooled crystals or  $\sim 125 \times 10^{-6}$  at  $\sim 11$  at. % Mo for quenched crystals. A sharp decrease after the peak is observed for the slow cooled crystals due to the formation of a second phase. The rhombohedral magnetostriction  $(3/2)\lambda_{111}$  of the Fe-Mo alloys is found to be insensitive to the Mo content. This behavior is distinctly different from other Fe-X ( $X=\text{Al}$ , Ga, and Ge) alloys where a slight decrease in magnitude and a sign reversal upon chemical ordering was observed for  $(3/2)\lambda_{111}$ . Both shear elastic constants ( $c'$  and  $c_{44}$ ) for Fe-Mo are remarkably insensitive to the Mo content, which is also distinct from the other Fe-based alloys used in the comparison. The two magnetoelastic coupling constants  $-b_1=3\lambda_{100}c'$  (with values from 7.15 to 9.77 MJ/m<sup>3</sup>) and  $-b_2=3\lambda_{111}c_{44}$  (with values from -4.96 to -5.81 MJ/m<sup>3</sup>) were calculated and compared with those of other Fe-X ( $X=\text{Al}$ , Ga, and Ge) alloys. © 2010 American Institute of Physics. [doi:10.1063/1.3359855]

## I. INTRODUCTION

The addition of nonmagnetic elements such as Al, Ga, and Ge into Fe enhances the tetragonal magnetostriction (MS) significantly. The tetragonal MS for Fe-Al,<sup>1,2</sup> Fe-Ga,<sup>3</sup> and Fe-Ge (Ref. 4) increases monotonically with the solute concentration in the bcc  $\alpha$ -Fe single phase region and reaches a maximum, which can depend on the thermal history, near the solubility limit. The large tetragonal MS in these Fe-X ( $X=\text{Al}$ , Ga, and Ge) alloys is a combination of an increase in the magnetoelastic coupling ( $-b_1$ ) and an extraordinary softening of the shear elastic constant  $c' = \frac{1}{2}(c_{11}-c_{12})$ .<sup>3,5</sup> Recently, Thuanboon *et al.*<sup>6</sup> investigated the MS and elastic properties of single crystalline Fe-Mo alloys. The tetragonal MS  $(3/2)\lambda_{100}$  was measured at 15 and 17.5 at. % Mo and the elastic moduli were determined at 15 at. % Mo. In contrast to the significant softening of  $c'$  observed for Fe-Ga alloys,<sup>5</sup>  $c'$  for the Fe-15 at. % Mo alloy was found to be the same as that of pure  $\alpha$ -Fe. The high tetragonal MS values observed in Fe-Mo, without the dramatic  $c'$  softening that accompanies other Fe-X alloys with X as groups 3 and 4 elements, suggest that a significant increase in the magnetoelastic coupling could be responsible for the observed behavior. Further, the compositions examined by Thuanboon *et al.*<sup>6</sup> were well beyond the room temperature solubility limit of Mo into the bcc  $\alpha$ -Fe structure. Little information is available for the alloys near and passed

the phase boundary. In this work we have systematically investigated the magnetoelastic behavior of single crystalline  $\text{Fe}_{100-x}\text{Mo}_x$  for  $2 < x < 12$ , a range which covers the transition from low to high solute concentration through the solubility limit. Further we have measured both shear moduli  $c'$  and  $c_{44}$  using resonant ultrasound spectroscopy and computed the magnetoelastic coupling behavior as a function of composition and thermal treatment.

## II. EXPERIMENT

Appropriate quantities of electrolytic grade iron (99.99% purity) and electronic grade molybdenum (99.99% purity) were arc melted several times into buttons under an argon atmosphere. Single crystal growth was done using the Bridgman technique in a resistance furnace.<sup>3</sup> Following crystal preparation, the ingots were annealed at 1000 °C (1350 °C for Fe-11 at. % Mo to be within the single A2 phase region at that concentration) for 168 h to homogenize the composition. Further, the crystals were cooled at 10 °C/min in a furnace filled with high purity argon. Oriented (100) and (110) disks, 6 mm in diameter and 2–3 mm thick, were sectioned from the ingot and marked with in-plane [100] and [111] directions for strain gage alignment, respectively. MS was measured by rotating the instrumented disk in a 20 kOe magnetic field applied parallel to the faces of the disk. The resulting strain was fitted to the lowest order MS term which follows a  $\cos^2 \theta$  angular dependence. The next higher order MS term, which follows a  $\cos^4 \theta$  angular dependence, was

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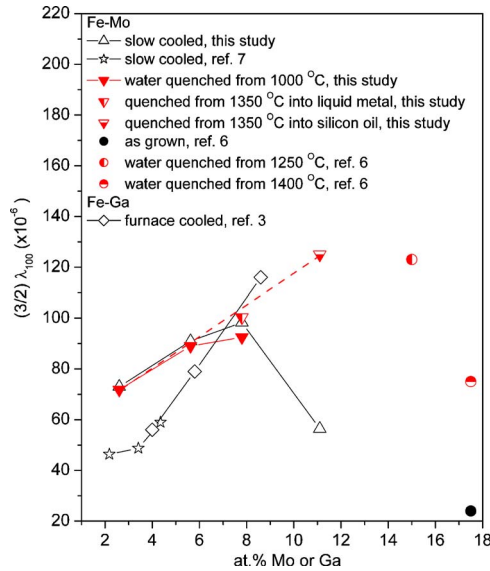


FIG. 1. (Color online) Tetragonal MS  $(3/2)\lambda_{100}$  of  $\text{Fe}_{100-x}\text{Mo}_x$  single crystals in both SC and quenched states compared with the data reported in the literature. The lines are guides to the eye.

generally less than 2 ppm. The MS measurements were repeated for the same disks after they were heat treated under one of three conditions (reheated at 1000 °C for 4 h and quenched into water, reheated at 1350 °C for 4 h and quenched into silicon oil, or liquid metal bath of In and Ga mixture). Single crystal samples in the shape of rectangular parallelepipeds (millimeter size, polished with 1  $\mu\text{m}$  diamond paste) with faces oriented along (100) were prepared for elastic constants measurements. Resonant ultrasound spectroscopy<sup>5</sup> was used to determine the two independent shear moduli of the crystals  $c'$  and  $c_{44}$  as a function of composition and thermal treatment. MS and elastic constants are the two variables upon which the magnetoelastic coupling constants  $b_1$  and  $b_2$  depend. The elastic constants measurements were done in a saturating magnetic field of 15 kOe oriented along the longest [100] axis of the parallelepiped. Composition measurements were done by energy-dispersive spectroscopy in a JEOL 840A scanning electron microscope (SEM).

### III. RESULTS AND DISCUSSION

Figure 1 compares the measured tetragonal MS  $(3/2)\lambda_{100}$  with literature data.<sup>3,6,7</sup> These results show that (i) for slow cooled (SC) crystals,  $(3/2)\lambda_{100}$  increases monotonically with Mo concentration, reaching  $\sim 100$  ppm at  $x \sim 8$  at. %; the tetragonal MS is about 40% higher than that reported by Hall<sup>7</sup> at similar compositions; a larger MS value than that of Fe–Ga (Ref. 3) is seen at low Mo content (less than 6 at. %); MS decreases sharply beyond 8 at. %. (ii) For the Fe–Mo crystals in the quenched state, MS increases monotonically with Mo concentration, reaching  $\sim 125$  ppm at  $x \sim 11$  at. %. It is observed that the thermal treatment affects the MS significantly but with a different behavior for low and high Mo concentration. For  $x < \sim 8$  at. %, the MS of the alloys quenched into water from 1000 °C is slightly lower than that of the SC ones. For  $x \geq 8$  at. %, the MS of

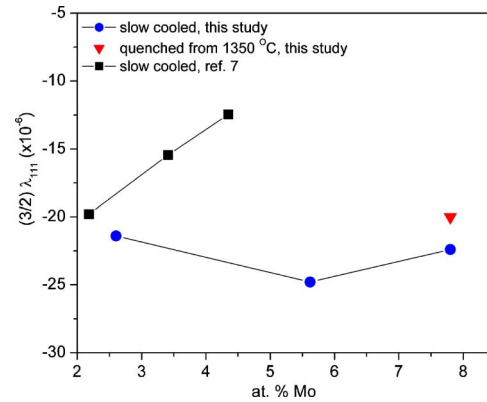


FIG. 2. (Color online) Rhombohedral MS  $(3/2)\lambda_{111}$  of  $\text{Fe}_{100-x}\text{Mo}_x$  single crystals in both SC and quenched states compared with the data reported in the literature.

the alloy in the quenched state is larger than that in the SC state. A significant difference is observed for the  $\text{Fe}_{88.9}\text{Mo}_{11.1}$  alloy, where the MS has more than doubled (from 56 to 125 ppm) by quenching the crystal into silicon oil from 1350 °C.

The dependence of the tetragonal MS on composition in Fe–Mo alloys follows a similar trend with that observed for Fe–Ga.<sup>3</sup> MS increases with Mo content up to a maximum that has been shown to correlate well with the solubility limit of the alloying element in the bcc Fe phase.<sup>8</sup> Once the solubility limit has exceeded, the formation of a second phase causes a marked and sharp drop in MS.<sup>3,4</sup> In systems where the solubility varies with temperature, thermal history has a significant effect on MS for alloys with a solute concentration near or above the solubility limit. Quenching from high temperatures may prevent or delay the formation of the second phase.<sup>9</sup> In Fe–Mo alloys, the formation of the second phase ( $\text{Fe}_2\text{Mo}$ ) has been confirmed by SEM microstructural analysis in both SC  $\text{Fe}_{92.2}\text{Mo}_{7.8}$  and  $\text{Fe}_{88.9}\text{Mo}_{11.1}$  crystals. An increase in the volume fraction of  $\text{Fe}_2\text{Mo}$  precipitates with increasing Mo content was observed.

Shown in Fig. 2 is the rhombohedral MS  $(3/2)\lambda_{111}$  for the  $\text{Fe}_{100-x}\text{Mo}_x$  alloys studied. In contrast to Hall's<sup>7</sup> results, where  $(3/2)\lambda_{111}$  decreases (in absolute value) with composition, our values are slightly larger in magnitude and do not change significantly with the Mo content. The rhombohedral MS was measured in both SC and quenched states for the  $\text{Fe}_{92.2}\text{Mo}_{7.8}$  crystal with no significant variation being found. An important feature of  $(3/2)\lambda_{111}$  for Fe–Mo alloys is that a sign reversal at the solubility limit, observed in both Fe–Ga (Ref. 3) and Fe–Ge (Ref. 10) alloys, was not found.

Table I lists the room temperature values of the two shear moduli  $c'$  and  $c_{44}$  for the SC and water-quenched (WQ)  $\text{Fe}_{100-x}\text{Mo}_x$  alloys and for SC pure  $\alpha$ -Fe. Both  $c'$  and  $c_{44}$  are insensitive to the Mo content with values close to those of pure  $\alpha$ -Fe. Quenching did not produce notable changes in the moduli. The value of  $c'$  reported by Thuanboon *et al.*<sup>6</sup> for quenched Fe-15 at. % Mo is 48.0 GPa. While this behavior has been observed before for  $c_{44}$  of Fe–Ge (Ref. 10) and Fe–Ga,<sup>5</sup> the pronounced softening of  $c'$  seen in the aforementioned alloys was not observed for  $\text{Fe}_{100-x}\text{Mo}_x$  in the composition range of  $2 < x < 9$ .

The magnetoelastic coupling constants were calculated



TABLE I. Elastic constants of pure Fe and Fe<sub>100-x</sub>Mo<sub>x</sub> single crystals in SC and WQ states measured at room temperature.

x (at. % Mo)	c' (GPa)		c <sub>44</sub> (GPa)	
	SC	WQ	SC	WQ
0	48.6		117.9	
2.7	49.0	49.0	115.9	117.6
5.6	49.4	48.4	117.2	116.4
8.5	49.9	50.8	114.5	114.4

using the measured MS and elastic constants according to the formulas  $-b_1 = 3\lambda_{100}c'$  and  $-b_2 = 3\lambda_{111}c_{44}$ . Since strains and elastic constants were measured at different compositions, interpolations were used to determine the value of the elastic constants at compositions where strains were measured. The calculated results for  $-b_1$  and  $-b_2$  are listed in Table II. Figure 3 shows  $-b_1$  for Fe<sub>100-x</sub>Mo<sub>x</sub> compared with other Fe-X (X=Al, Ga, and Ge) alloys<sup>1,5,10</sup> (all in the SC state).<sup>5,10</sup> The magnetoelastic coupling factor  $-b_1$  increases monotonically from  $\sim 7$  MJ/m<sup>3</sup> at  $x=2.6$  to  $\sim 10$  MJ/m<sup>3</sup> at  $x=7.8$ . These values are slightly larger than those for Fe-Al,<sup>1</sup> Fe-Ga,<sup>5</sup> and Fe-Ge (Ref. 10) at similar compositions. It can also be seen that  $-b_1$  of Fe<sub>100-x</sub>Mo<sub>x</sub> does not show a maximum at the solubility limit ( $x \sim 5$  at. % Mo), as was found in the other Fe-X alloys. Similarly,  $-b_2$  follows the trend of  $(3/2)\lambda_{111}$  because  $c_{44}$  is insensitive to the Mo content and does not change sign at the solubility limit as observed in Fe-Ga (Ref. 5) and Fe-Ge.<sup>10</sup>

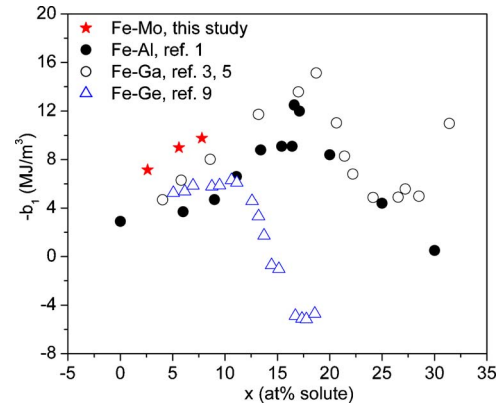
The origin of the increase in magnetoelasticity with Mo additions to  $\alpha$ -Fe appears to be different from those found with Al, Ga, and Ge additions. The high tetragonal MS values observed in Fe-Mo occur without the dramatic  $c'$  softening that accompanies other Fe-X alloys, where X are groups 3 and 4 elements. This suggests that the enhanced coupling is solely due to different ground-state electronic structures. It is known that spin-orbit coupling interaction between the occupied and unoccupied states can play a critical role for determining the MS in Fe-X alloys.<sup>11</sup> While the d-shell is fully occupied in both elemental Ga and Ge, the d-shell is half-filled in elemental Mo. First principles calculations are ongoing to determine if similar electron interactions are present in Fe-Mo alloys.<sup>12</sup>

#### IV. SUMMARY

The magnetostrictive and elastic properties for single crystalline Fe<sub>100-x</sub>Mo<sub>x</sub> alloys with  $2 < x < 12$  were investigated. The tetragonal MS of both SC and quenched Fe-Mo alloys follows similar trends to other Fe-based alloys. The tetragonal MS increases with Mo composition to a maximum

TABLE II. Magnetoelastic coupling constants  $-b_1$  and  $-b_2$  for SC Fe<sub>100-x</sub>Mo<sub>x</sub> measured at room temperature.

x (at. % Mo)	2.6	5.6	7.8
$-b_1$ (MJ/m <sup>3</sup> )	7.15	8.99	9.77
$-b_2$ (MJ/m <sup>3</sup> )	-4.96	-5.81	-5.17

FIG. 3. (Color online) Comparison of room temperature magnetoelastic coupling constants  $-b_1 = 3\lambda_{100}c'$  vs  $x$  for Fe<sub>100-x</sub>Mo<sub>x</sub> and Fe<sub>100-x</sub>X<sub>x</sub> (X=Al, Ga, and Fe) alloys.

and then decreases due to the formation of a second phase. Quenching the crystals from the high-temperature A2 phase region increases the MS for alloys with a Mo composition beyond the room temperature solubility limit. The rhombohedral MS is insensitive to the Mo content in Fe-Mo alloys. Both shear elastic constants ( $c'$  and  $c_{44}$ ) for Fe-Mo are similar to those of pure  $\alpha$ -Fe, being insensitive to the Mo content. No softening in  $c'$  observed for other Fe-X (X=Al, Ga, and Ge) alloys was seen for Fe-Mo. The two elastic coupling constants  $-b_1 = 3\lambda_{100}c'$  and  $-b_2 = 3\lambda_{111}c_{44}$  were calculated and were found to be larger than other Fe-X (X=Al, Ga, and Ge) alloys at similar compositions.

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